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A. CORMAN

N. J. GUARINO

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WASHINGTON, D. C.

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CANISTER FOR PRODUCING TMA TRAILS IN
THE UPPER ATMOSPHERE

A. Corman

N. J. Guarino

February 1965

Contract No. NASw-1083

GCA CORPORATION
GCA TECHNOLOGY DIVISION
Bedford, Massachusetts

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CANISTER FOR PRODUCING TMA TRAILS IN
THE UPPER ATMOSPHERE

By A. Corman and N. Guarino
GCA Corporation

SUMMARY

20793

A trimethylaluminum (TMA) payload canister, which is suitable for installation on the Nike Cajun or Apache rocket vehicles, was designed, built, and flight tested within a ten-week period. The first test flight, Nike Apache 14.145 launched from Wallops Island, Virginia on 7 October 1964, was completely successful. Release of the TMA was initiated as programmed at 251 seconds after lift-off and a visible trail was generated. Since the TMA module is interchangeable with the GCA Corporation's sodium vaporizer either chemical can be flown with the Langmuir probe.

Major difficulties encountered during the development program concerned payload safety, handling, and shipping of the TMA canister. The material is classified as "Red Label Shipment" by the Interstate Commerce Commission (ICC) and must be transported in ICC approved containers. ICC permit No. BA-464 was received for the approved container. Safety and handling procedures were specified for personnel who would service the payload.

The canister design consists basically of a seamless teflon bladder which contains the TMA, a pressurizing source and explosively actuated valve with a flow-control orifice, a programmer, and an external shell. Primary design features are:

(1) High-expulsion efficiency: 99% of the stored liquid is expelled by the bladder.

(2) Independence of vehicle motions: the method of expulsion, that is squeezing the bladder by means of a constant pressure, forms a solid jet which is not affected by vehicle motions. For example, a uniform jet would be obtained even if the vehicle were tumbling.

(3) Safe handling and storage: the use of teflon for the bladder material permits a long storage life. Redundant seals are used; the bladder serves as the primary seal and "O"-rings seal the external metal shell.

(4) Safe and reliable filling methods: the canister is filled in accordance with approved filling procedures at the Chemical plant (Texas Alkyls, Pasadena, Texas).



INTRODUCTION

The development of a rocket payload module, which will release a trail of TMA* in the upper atmosphere for measurement of winds, is described in this Technical Report. Chemical reactions, which occur above 80 km, between the TMA and the ambient atmosphere create a chemiluminescent cloud which forms a trail owing to the rocket motion. Ground observations of the trail enable the observer to determine wind patterns. The TMA module was designed to fly with a Langmuir probe which measures electron density.

The chemical dispenser is the first of its kind to have been mated with an instrumented payload for the scientific purpose of correlating wind patterns and electron density. Combining the chemical instrumented payload modules was suggested by Mr. Maurice Dubin of the National Aeronautics and Space Administration.

Figure 1 shows the combined electron density TMA module payload, partially disassembled; the instrumentation rack which programs the TMA release is shown. Figure 2 shows the assembled payload that was successfully flown.

HISTORY OF DEVELOPMENT

Background Information

Previous rocket-borne TMA payloads have been designed and built by Air Force Cambridge Research Laboratories and Sandia. Their design approaches were assessed with the intent of forming a sound basis for the present design. Neither design appeared to satisfy the requirements of reliable operation, high efficiency, and safe handling. It was decided therefore that the GCA Corporation design would employ a different design approach which would satisfy a number of requirements.

Design Requirements

The chemical dispenser was to be combined with a Langmuir probe payload in order to correlate winds and electron density. Since the effects of TMA release on the operation of the probe were not known, Mr. Dubin of NASA (program director) suggested that the electron probe function up to apogee at which time the TMA would be expelled. Thus electron density would be obtained on ascent and winds on descent. Based on the Nike Apache trajectory, it was determined that a minimum trail release time of 90 seconds would be required.

*A mixture of 75% TMA and 25% TEA (triethylaluminum) is used. The TEA serves as an anti-freeze. Chemical data is given in the appendix.

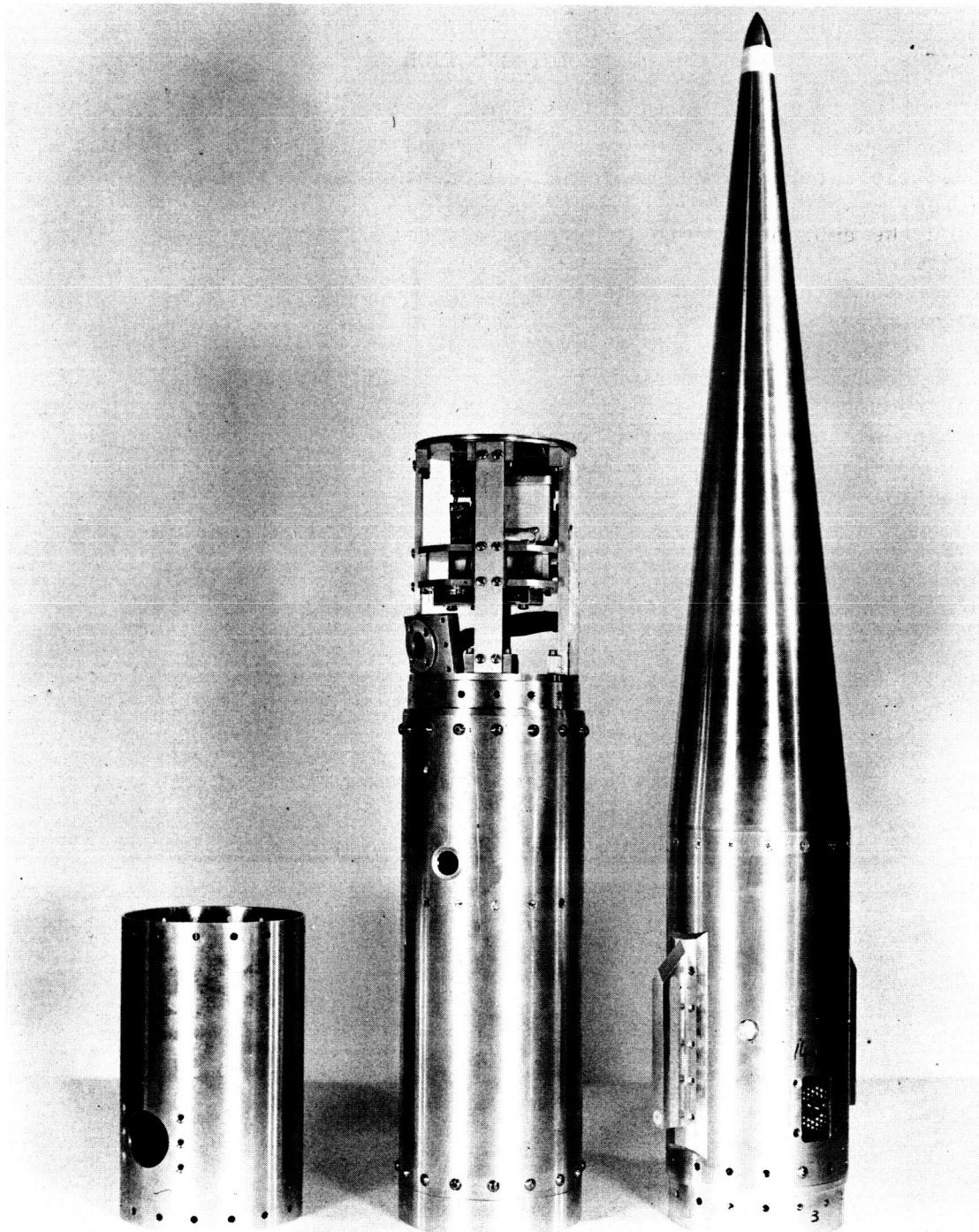


Figure 1. Combined electron density - TMA. Module Partially disassembled.

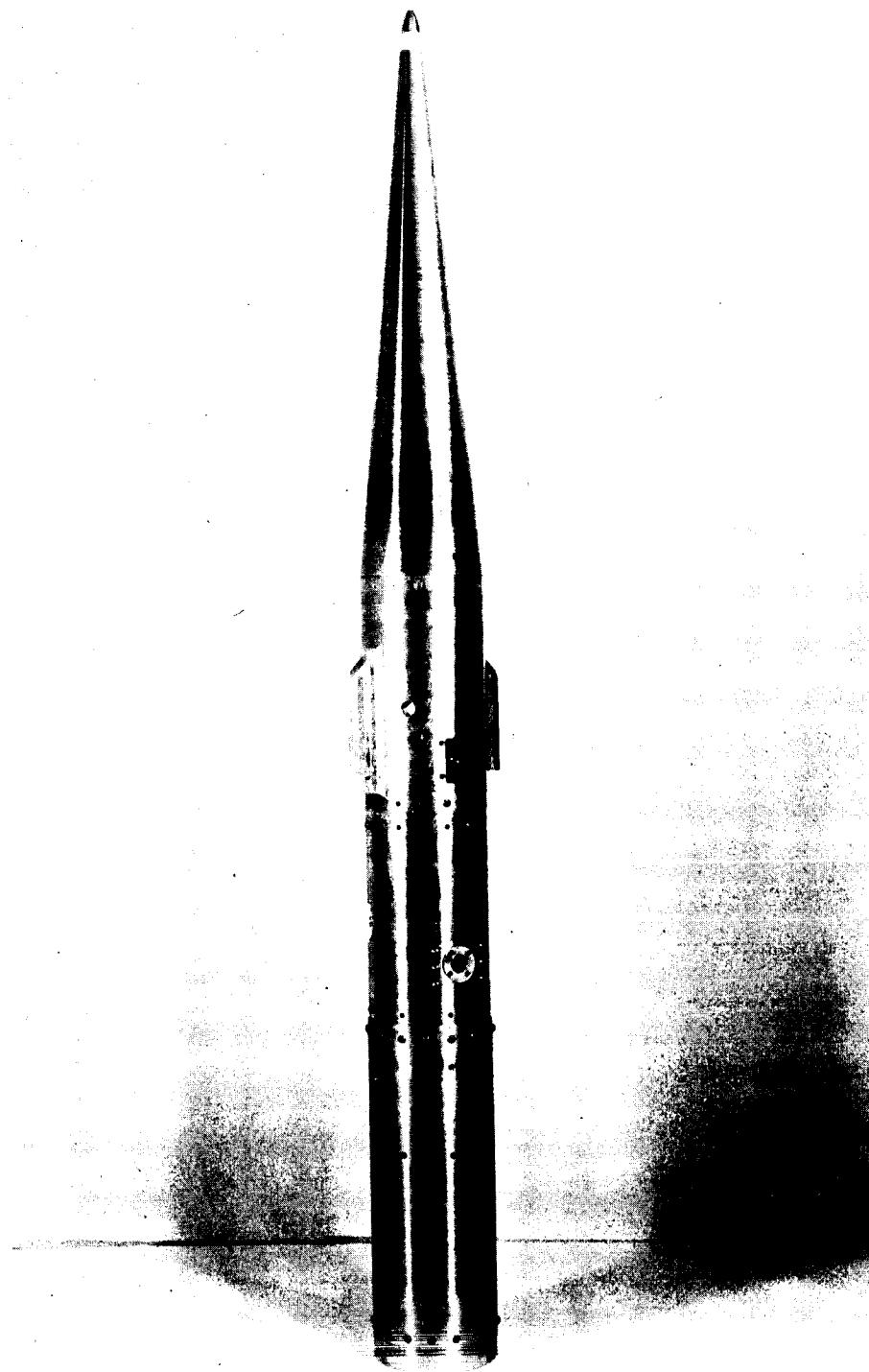


Figure 2. Combined payload, assembled.

Design requirements for the TMA payload module were as follows: (1) produce a trail having 90 seconds minimum duration; (2) canister must be electrically and mechanically interchangeable with the existing sodium canister; (3) canister must contain a maximum quantity of TMA in a volume about the same as the existing sodium canister; (4) canister must be ICC approved to allow loading at the chemical plant and shipment via common carrier to launch site; (5) expulsion characteristics must be independent of vehicle dynamics; (6) design must use standard components owing to the short time available; and (7) the canister must be safe to handle and have maximum storage life.

Development Schedule

The short time available to develop the TMA canister, in order to meet previously scheduled launch dates, meant that delivery dates of materials and components would play an important part in the design considerations. A launch date of 30 September 1964 was specified for flight of the first payload. There was not sufficient time for the usual development cycle of "design - build - test - modify - fly"; the first approved paper design was fabricated and flight tested.

The development schedule which was programmed at the start is shown in Figure 3. A project team, which consisted of a project engineer, two design engineers, and two technicians, was formed and devoted full-time to this task. A high priority was assigned to the project with work to proceed on a "non-interruption" basis.

Continual liaison was maintained with the ICC, Texas Alkyls (chemical manufacture), and vendors supplying long-lead time items. Personal conferences were held with ICC officials in New York City, and the module design was thoroughly reviewed. Several design changes recommended by the ICC for safety purposes were incorporated. Approval from the ICC was received on 18 August 1964 after which time the manufacturing drawings were released for fabrication.

The first unit was assembled on 4 September 1964. Expulsion tests were run, under room conditions, to determine orifice size for the desired flow time. Based on 25 psi bladder pressure, an orifice size of 0.059 in. was chosen which resulted in 150 seconds of flow.

Assembly procedures for the canister were investigated. Owing to the interference fit of the teflon bladder in the shell, extreme care was necessary for its insertion. In one instance, pressure checks indicated a ruptured bladder. Disassembly showed that the bladder was pinched and severed in one area. The assembly procedure was reviewed and the bladder was inserted in a collapsed condition with the bottom end cap (payload adapter) removed. This allowed observation of the bladder clearing the internal diameter of the shell.

Another development problem which arose during bench testing of the canister was the sealing of the bladder flange between the upper bladder support

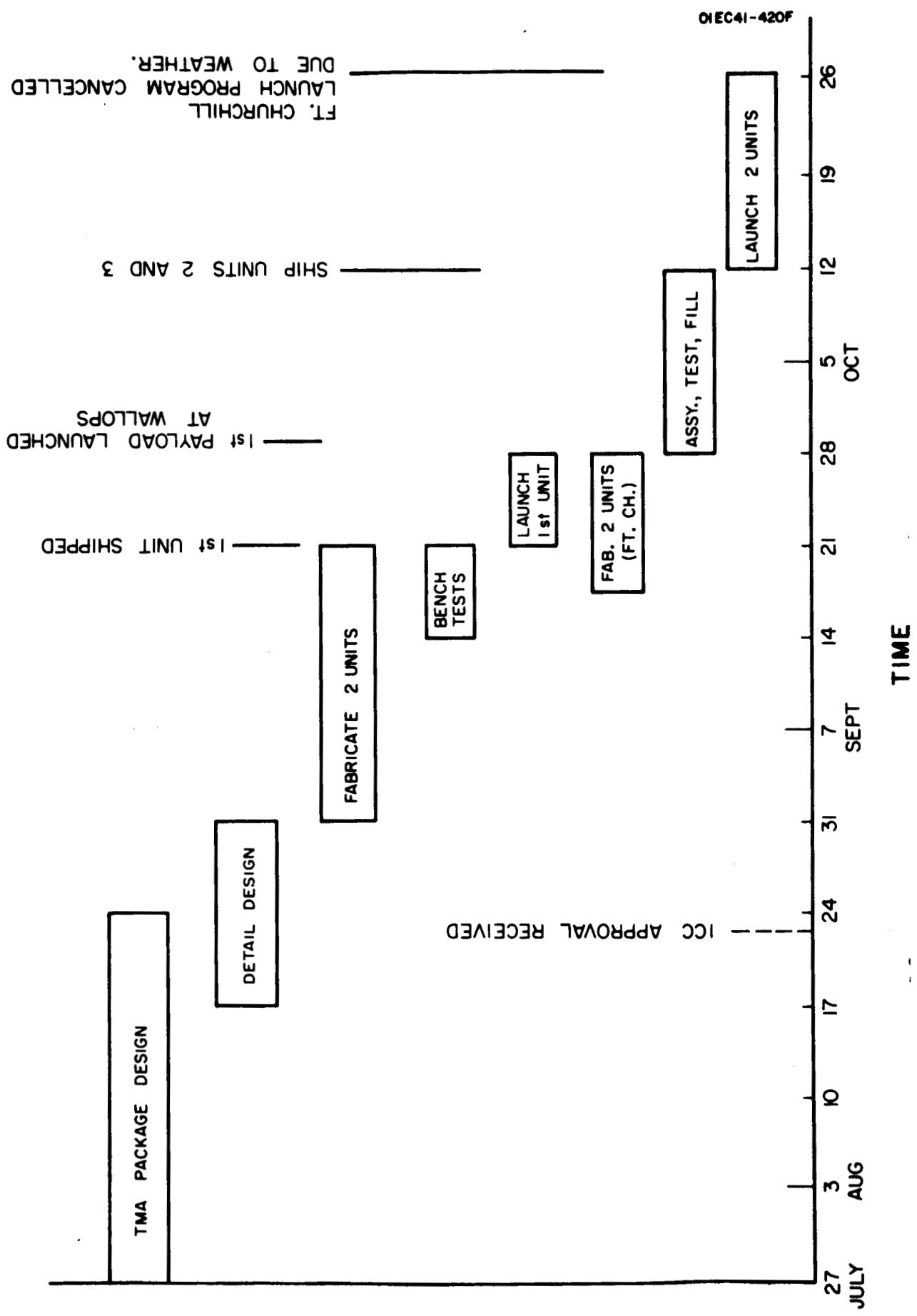


Figure 3. Development schedule.

and valve housing. It was found that slight variations in the thickness of the flange caused unequal compression as the flange was sandwiched between the two metal parts. This problem was solved by machining a small circular ridge on the valve housing which tightly compresses the flange along a circle which is concentric with the bolt circle in the flange. Pressure tests were run and no further sealing problems were experienced and no further development problems were encountered.

Description of Module

The TMA module consists of a flexible expulsion bladder, valve assembly, pressure vessel, canister shell, and seals. Figure 4 shows the general configuration of the module, and Figure 5 is the assembly drawing.

Approximately 6 pounds (3.5 liters) of TMA is contained in the teflon bladder. Since it is completely supported by the metal shell the strains on the bladder during powered flight are eliminated. Dry nitrogen, regulated to 25 psi, is introduced between the bladder and shell. The pressure causes the bladder to collapse and expel the chemical out of the module canister through an orifice. For a given pressure the orifice size determines the flow rate of the chemical. Since the pressurizing gas is not in direct contact with the chemical liquid, the gas cannot form bubbles in the expellant and escape through the orifice. Hence, use of the bladder assures a continuous solid stream which is independent of vehicle spin, coning, and attitude.

An O-ring seal at each end of the canister seals the exterior of the bladder. These seals serve two purposes, first they seal the space between the bladder and the shell which allows pressurization and second to prevent leakage from the canister if a leak should develop in the teflon bladder wall. These seals are made of VITON A which has good resistance to attack by TMA. Kel-F elastomer seals are slightly superior to VITON A with respect to corrosion resistance, however, these seals could not be obtained in time for the first test flight. Details of the basic components are discussed in the following paragraphs.

Expulsion bladder. - The specifications of the teflon bladder, Figures 6 and 7, are based on the bladder manufacturers proven design with an established reliability of 0.99999 for at least 50 cycles of filling and complete expulsion by collapsing with external gas pressure. To assure high reliability it is necessary to mount the bladder so that the teflon is not subjected to any tensile forces which would stretch it and cause leaks. This requires careful consideration of all internal dimensions and tolerances of the supporting shell and end pieces. Dimensions and tolerances were selected to ensure that the shell would be slightly smaller in all directions than the bladder by .010 in. to .030 in. The mouth of the bladder is sealed when the teflon flange is sandwiched between the upper bladder support which is shown in Figure 7 and the valve housing. These pieces are fastened by six Absco No. AB1533-f-7 self-sealing screws. The sealing surface of the valve housing has raised concentric rings which bite into the teflon bladder flange and assure a leakproof seal when the self-sealing screws are made tight.

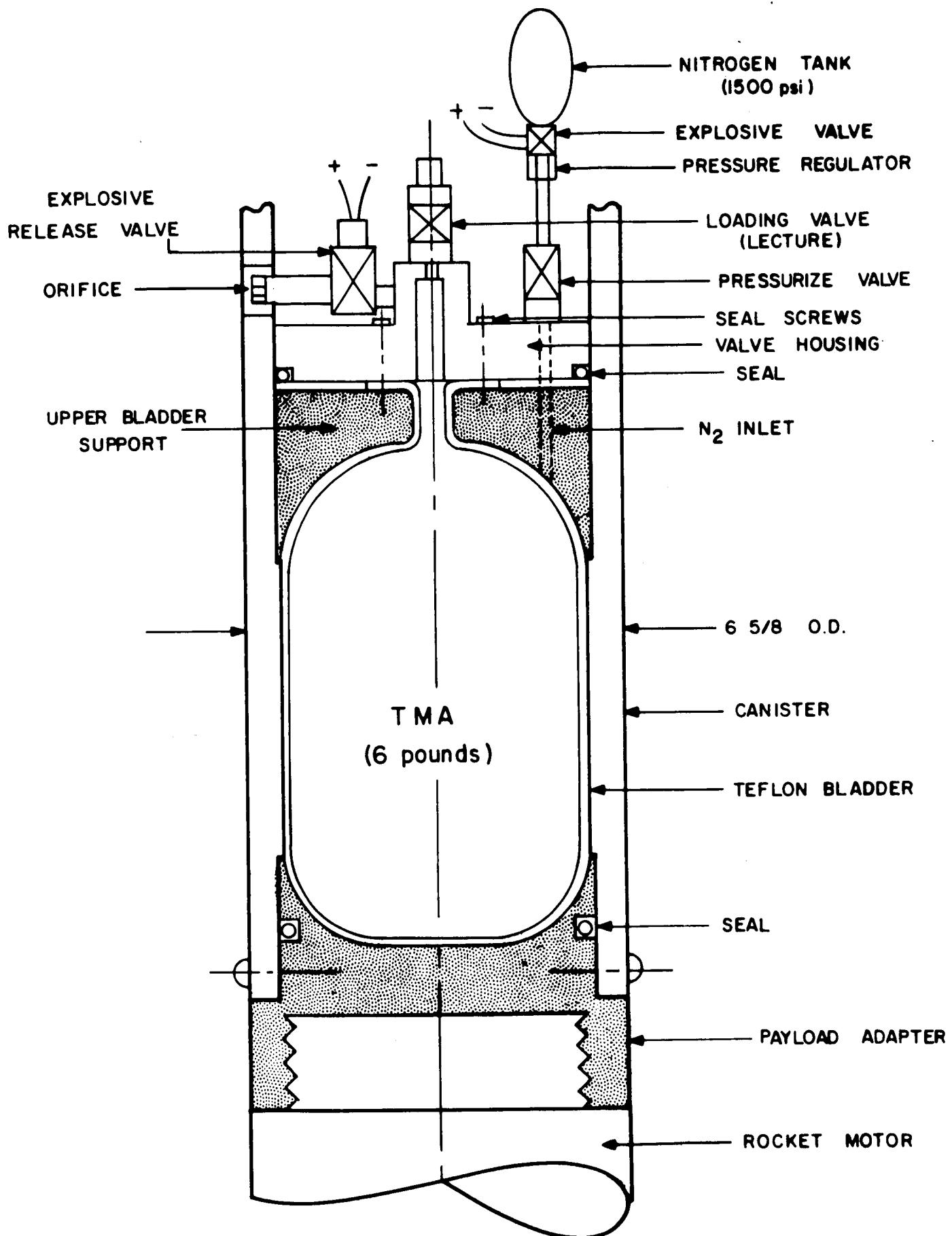
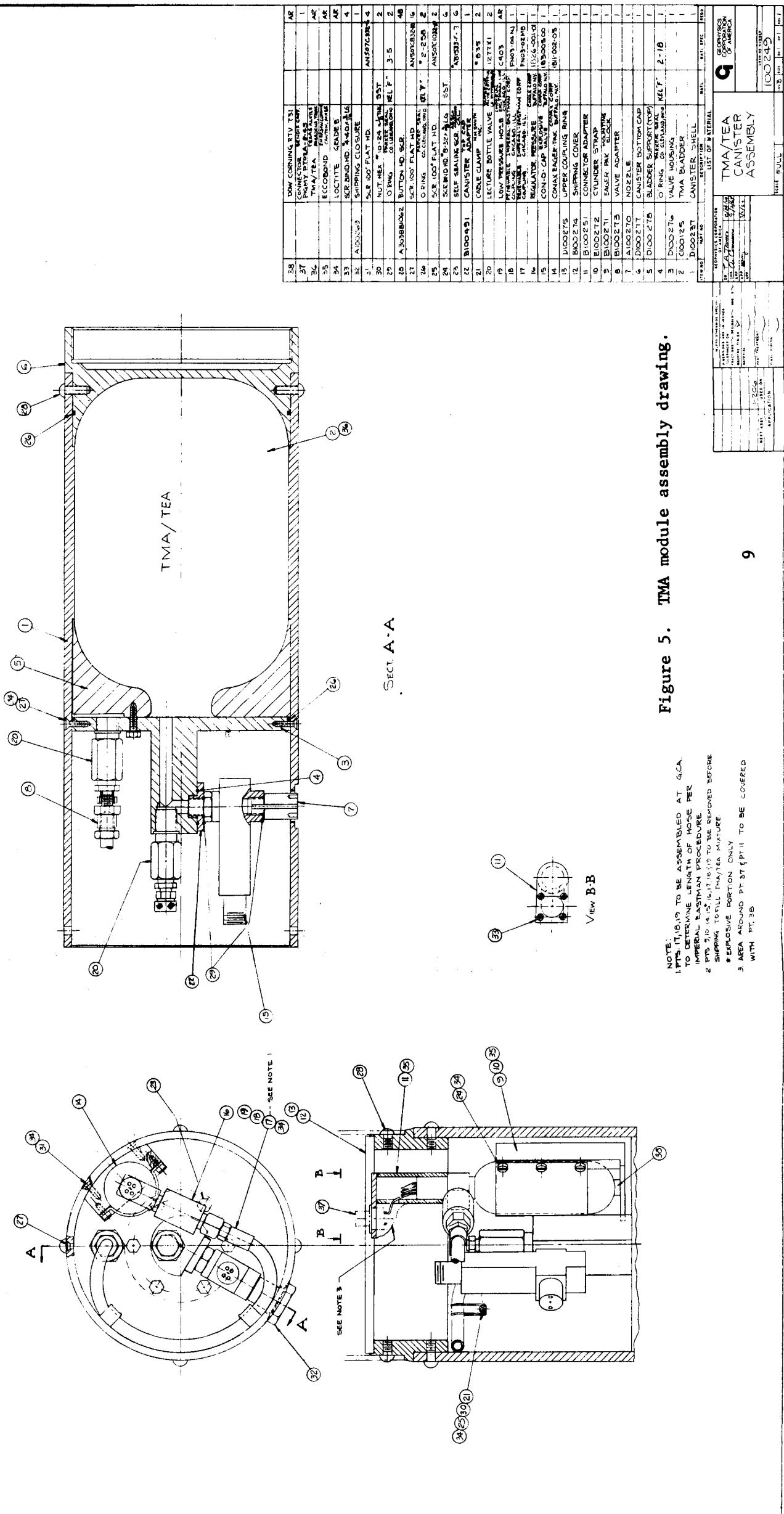


Figure 4. TMA payload module for Nike Cajun/Apache.

1	SET ECO FCS	1	SET ECO FCS
2	IMPERIAL LASTMAN PROCEDURE	2	IMPERIAL LASTMAN PROCEDURE
3	AREA AROUND PT. 37 1/2 IN. TO BE REMOVED BEFORE FILLING TO FULL TMA/TEA MATURE * EXPLOSIVE PORTION ONLY WITH PT. 35	3	AREA AROUND PT. 37 1/2 IN. TO BE REMOVED BEFORE FILLING TO FULL TMA/TEA MATURE * EXPLOSIVE PORTION ONLY WITH PT. 35
4	NOTE: 1. PTS. 7,16,19 TO BE ASSEMBLED AT G.C.A. TO DETERMINE LENGTH OF HOSE PER IMPERIAL LASTMAN PROCEDURE	4	NOTE: 1. PTS. 7,16,19 TO BE ASSEMBLED AT G.C.A. TO DETERMINE LENGTH OF HOSE PER IMPERIAL LASTMAN PROCEDURE
5	2. PTS. 9,10 14 1/2", 17 1/2" TO THE REMOVED BEFORE FILLING TO FULL TMA/TEA MATURE	5	2. PTS. 9,10 14 1/2", 17 1/2" TO THE REMOVED BEFORE FILLING TO FULL TMA/TEA MATURE
6	* EXPLOSIVE PORTION ONLY	6	* EXPLOSIVE PORTION ONLY
7	3. AREA AROUND PT. 37 1/2 IN. TO BE COVERED WITH PT. 35	7	3. AREA AROUND PT. 37 1/2 IN. TO BE COVERED WITH PT. 35

1. PTS. 7,16,19 TO BE ASSEMBLED AT G.C.A.
TO DETERMINE LENGTH OF HOSE PER
IMPERIAL LASTMAN PROCEDURE
2. PTS. 9,10
14 1/2", 17 1/2" TO THE REMOVED BEFORE
FILLING TO FULL TMA/TEA MATURE
* EXPLOSIVE PORTION ONLY
3. AREA AROUND PT. 37 1/2 IN. TO BE COVERED
WITH PT. 35

Figure 5. TMA module assembly drawing.



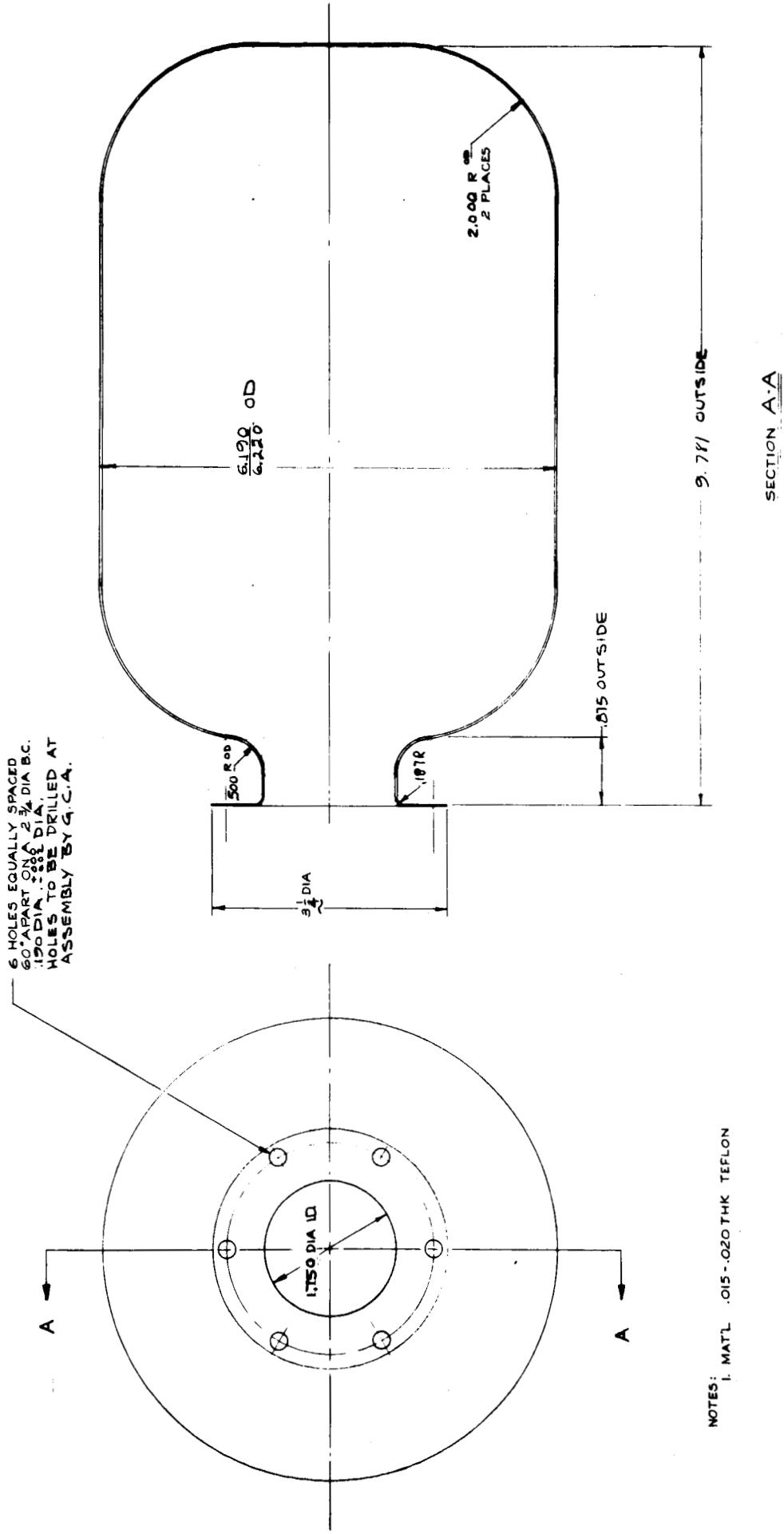


Figure 6. TMA teflon bladder.

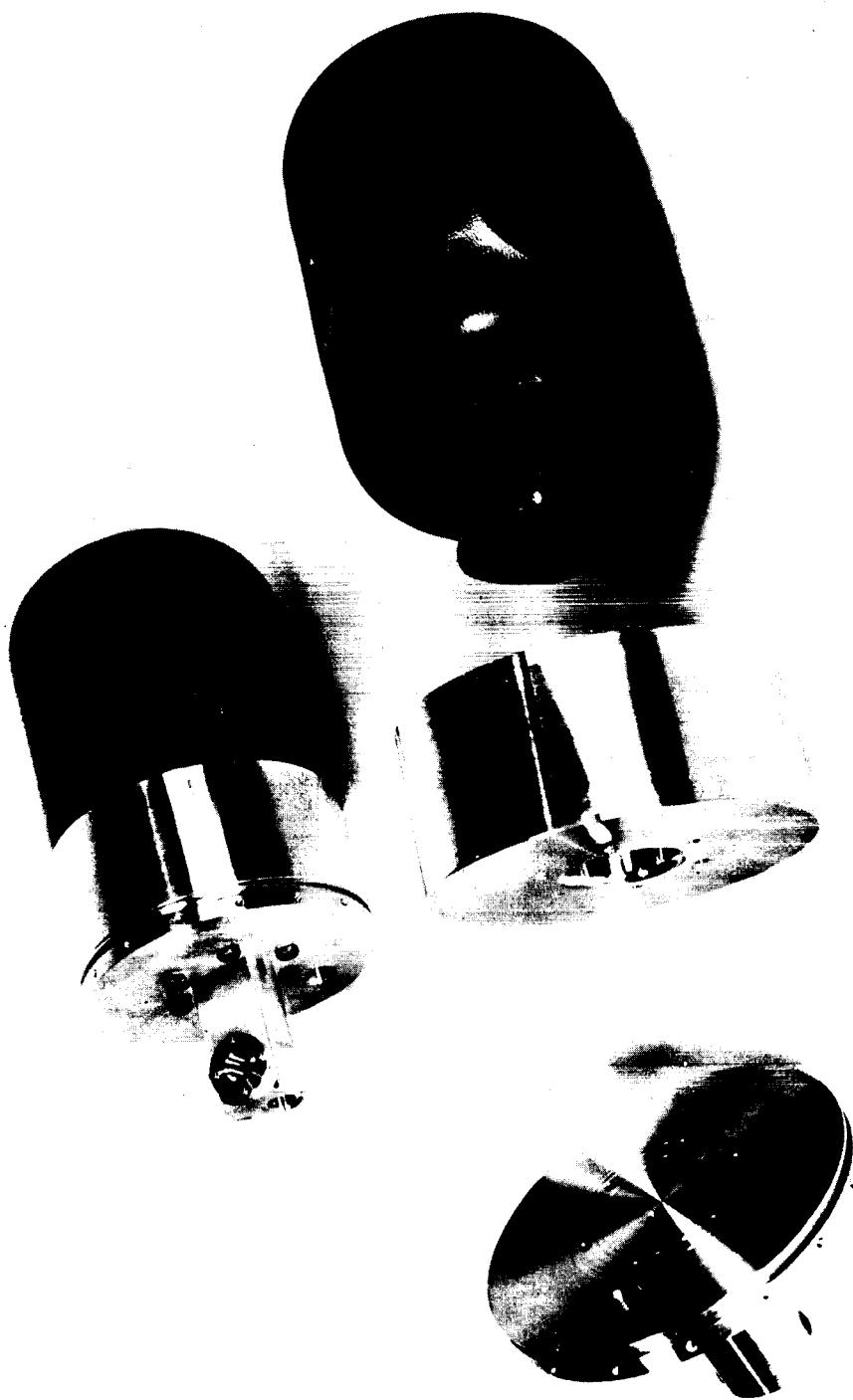


Figure 7. TMA bladder and mounting flange.

Valve assembly. - Three valves, mounted to the valve housing which is shown in Figure 8, are for chemical loading, pressurization, and chemical release. The loading valve, a brass lecture bottle valve with teflon seals, is mounted on top of the vertical projecting section of the valve housing and provides a path directly into the bladder for chemical loading. Connected into this same path is the explosively opened release valve. This valve is supplied by the Conax Corporation and has demonstrated high reliability in similar applications. The valve is activated when an electric current is passed through a bridgewire which fires a contained explosive charge. The explosive force drives a piston forward shearing a metal cap in the valve body and opening the valve. Since the metal cap is machined integral with the valve body, the valve is absolutely leak proof until sheared. These valves are factory proof tested at 7500 psi and have a bursting strength of 10,000 psi. The maximum operating pressure is 5000 psi. The operating time of this valve at the recommended firing current of 2 A is 2 msec. Since the flow control orifice screws into the valve outlet from outside the shell, orifice changes can be made on the loaded canister when attached to the vehicle. A pressurizing valve is mounted to one side of the valve housing and seals the path leading to the area between the bladder and the outer shell. The gas pressure for expelling the contents of the bladder is introduced through this valve. The loading and pressurizing valves (Superior No. 1277X1 lecture bottle valves) are hollow stem needle valves with a female pipe thread so that external plumbing can be connected at the top of the stem. The configuration is convenient for this application where access to the valves for external connections and operation must be made in limited space from the top as shown in Figure 9.

Pressurization. - The Conax Eager-Pak shown in Figure 8 is equipped with a Conax 25 psi pressure-regulator which provides the expulsion force. The Eager-Pak is a commercial component consisting of a 4.6 cu in. pressure vessel, containing about 450 cu in. STP nitrogen at 1500 psi; the vessel is fitted with an explosive actuated valve. The quantity of gas when emptied into the canister will result in a terminal pressure of about 25 psi when the bladder is fully collapsed. The Eager-Pak assembly is mounted to the inner wall of the upper portion of the canister shell above the valve housing. A rubber pressure hose connects the pressure regulator on the Eager-Pak to the pressurizing valve.

Canister Shell. - The canister shell, 6061-T6 aluminum, is 6-5/8 in. in diameter and 1/4 in. thick. The bottom cap provides the coupling thread for attaching the canister to the vehicle headcap.

The upper part of the shell is extended to protect the valves and pressure tank. The upper end is machined to accept the standard programmer and power supply presently used for the sodium vaporizer canisters supplied to NASA.

Testing

Preliminary expulsion tests were conducted using a bladder installed in a transparent plastic shell; thus observation of the entire expulsion process and the physical behavior of the bladder under dynamic conditions was possible.

410



Figure 8. Components of expulsion system.



Figure 9. TMA canister - top view.

The bladder was filled with water and compressed air supplied the expulsion force. The system was cycled 20 times. The bladder withstood the repeated crushing and reforming without developing any leaks. Figure 10 shows the bladder during the various stages of expulsion. Figure 11 shows the bladder during the various stages of filling.

All flight bladders were leak tested at 5 psi when received from the manufacturer before they were installed in the canisters.

A 100-psi bubble-leak test of the fully assembled canisters checked the O-ring and bladder flange seals. This is double the pressure required to meet the ICC requirements for a container normally operating at 25 psi. The test set-up is shown in Figure 12, and is conducted as follows:

With canister immersed in water

- (1) Open pressure and loading valves.
- (2) Close valves 1 and 2.
- (3) Adjust nitrogen regulator to 10 psi.
- (4) Open valve 2 pressurizing bladder to 10 psi.
- (5) Open valve 1 equalizing pressure on both sides of bladder.
- (6) Close valve 1.
- (7) Increase nitrogen regulator output another 10 psi.
- (8) Open valve 1 to equalize pressures.
- (9) Close valve 1.
- (10) Repeat steps 7, 8, and 9 until 100 psi is reached.

All seals and screw heads are observed for bubbling after each pressure rise. When the maximum pressure of 100 psi is reached, the canister is observed for at least 15 minutes for any signs of leakage. This procedure effectively tests all external canister and valve mounting seals and ensures that no pressurizing gas will be lost resulting in incomplete expulsion when using the limited gas volume available in flight. The reason for raising the pressure in 10-psi increments and equalizing the pressure outside the bladder is to prevent damage to the bladder from unnecessary wrinkling or stretching of the teflon. In normal operation, the maximum pressure differential across the bladder will not exceed 4 to 5 psi.

A further test, not illustrated, is to check for leakage between the bladder and the canister shell. This is done by pressurizing the bladder in the assembled unit to 15 psi through the loading valve. A hose is connected to the pressure valve with the open end immersed in water and observed for bubbling. Bubbles would indicate leakage at the bladder flange or through a hole in the bladder wall. A leak of this nature would not be apparent outside the canister because the outer seals would safely contain the TMA. It could, however, interfere with the expulsion process by allowing the pressurizing gas to mix with the TMA and be lost. The result would be incomplete expulsion and irregular flow.

Expulsion times vs orifice diameter at different pressures were determined by use of the set-up shown in Figure 13. The canister was filled with 3.5 liters

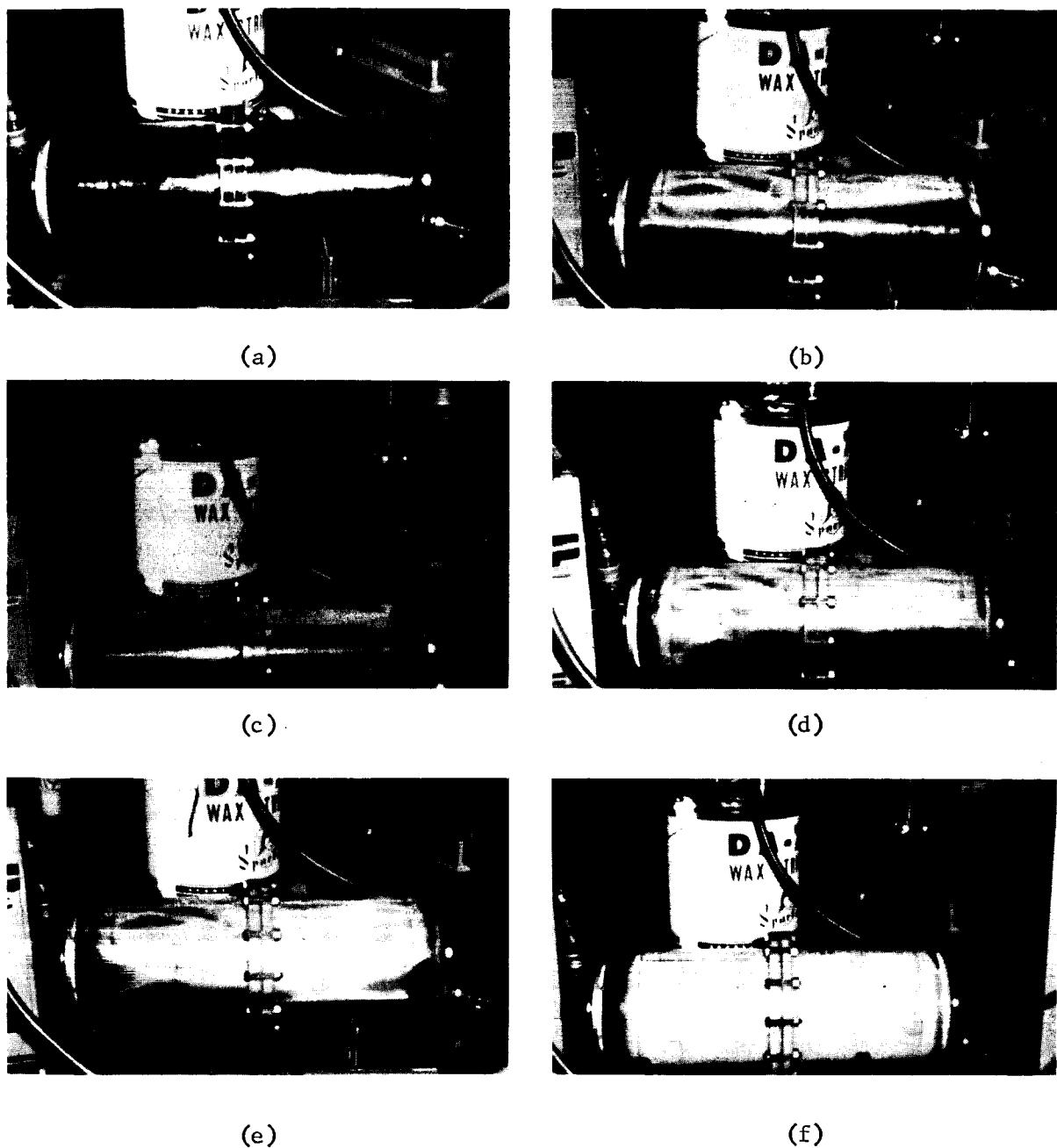


Figure 10. Bladder expulsion.

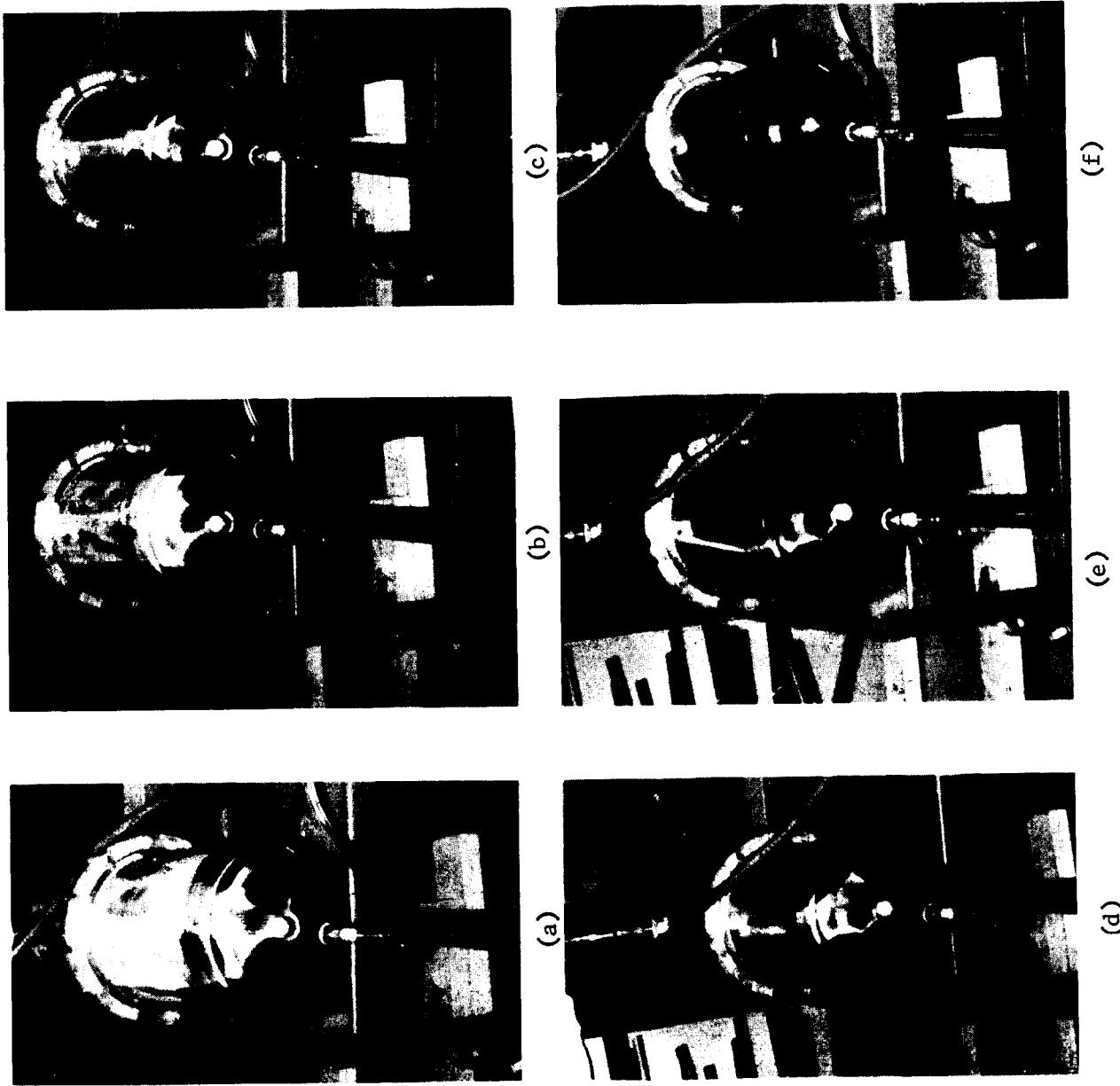


Figure 11. Bladder filling.

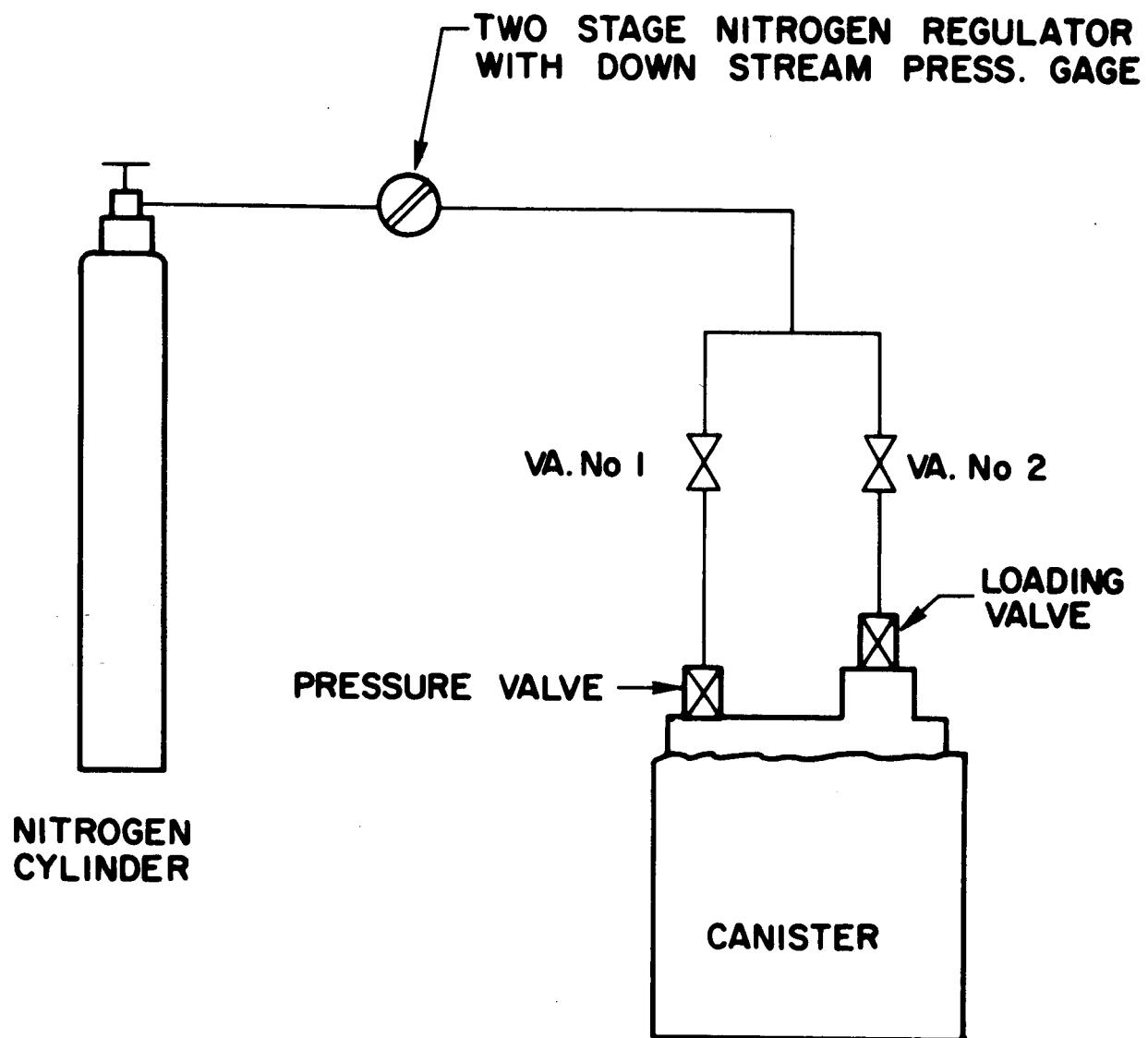


Figure 12. TMA canister pressure test set-up.

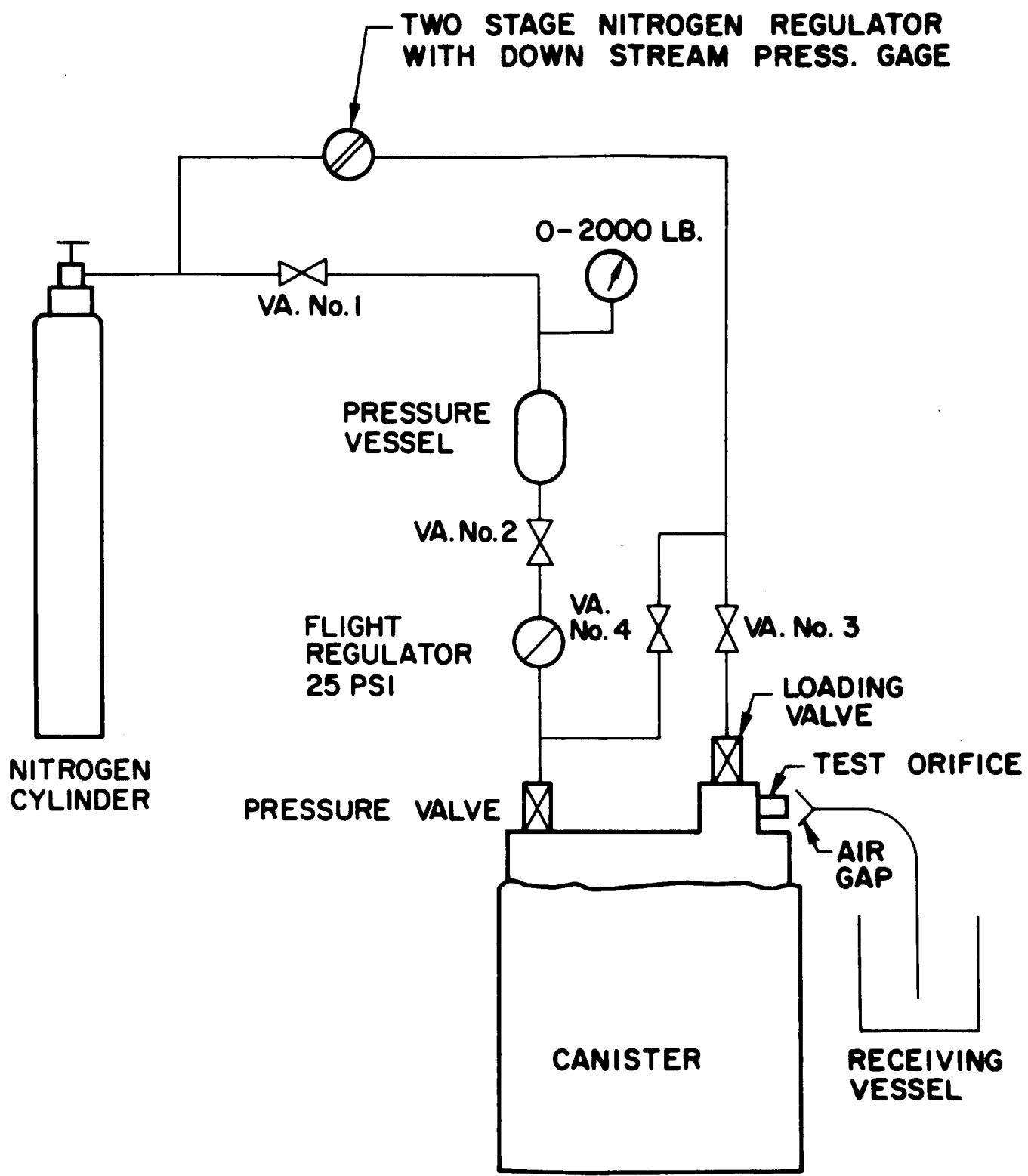


Figure 13. TMA canister flow rate calibration set-up.

of dry kerosene which is similar to TMA in specific gravity and viscosity. Two methods were used to pressurize the canister. The first used a constant pressure throughout the expulsion cycle. The second method simulated the actual flight conditions using a pressure vessel having the same volume and filling it to the same pressure as the Eager-Pak units used in flights. The contents of this vessel were expelled into the canister through the Conax flight regulator. The kerosene was expelled into a tank and the expulsion efficiency (ratio of amount expelled to the amount stored) was determined.

The tests were run using different orifice sizes resulting in the curves shown in Figure 14. Of the 3.5 liters in the bladder, 3.469 liters were expelled giving an expulsion efficiency of more than 99%.

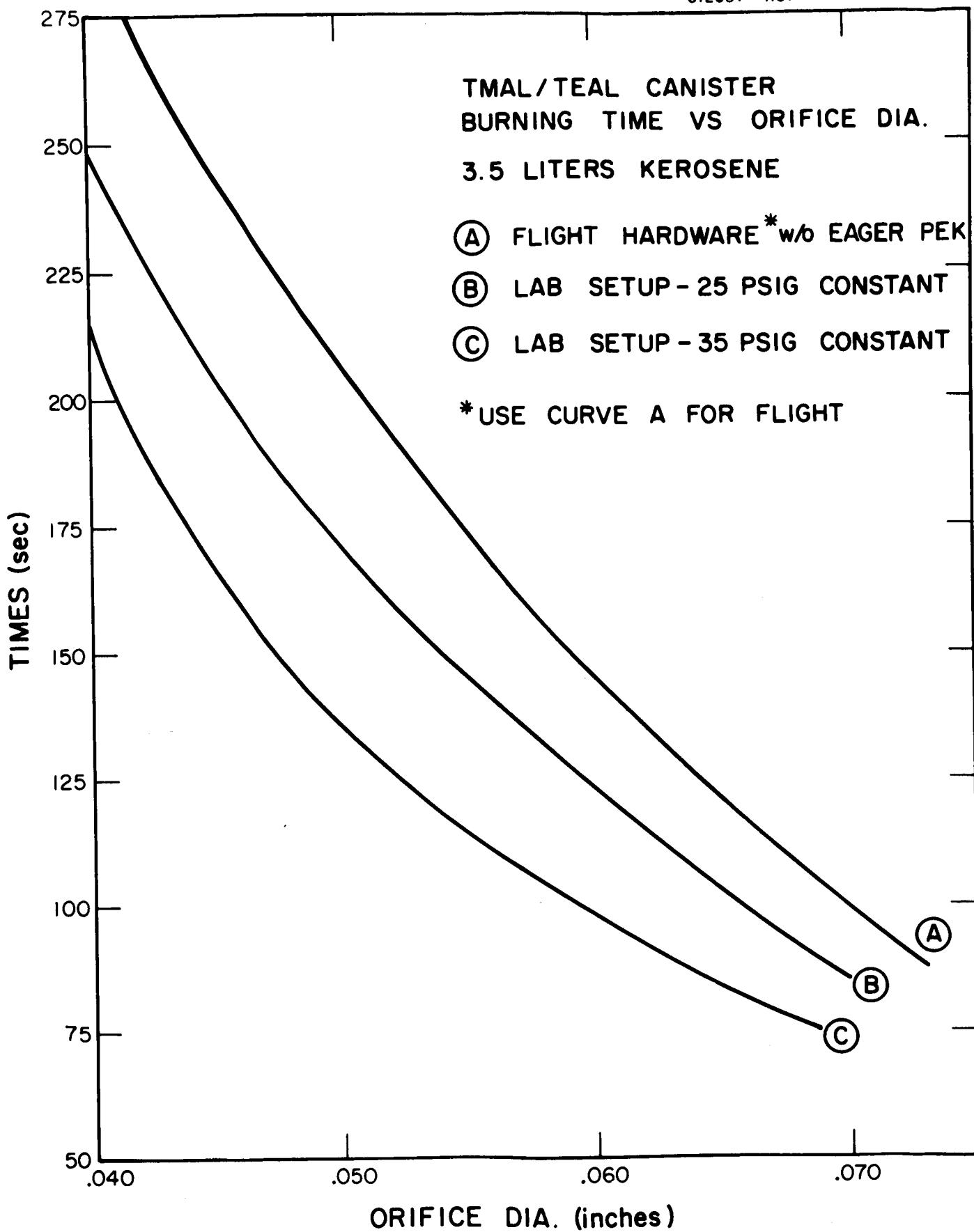


Figure 14. Time vs orifice diameter.

SHIPPING

The ability to ship the loaded canister from the chemical supplier to the test site was one of the prime design objectives. The Bureau of Explosives in New York supplied us with the pertinent requirements and procedures, as set forth in ICC regulation 7 3.134. The preliminary design layout was made using the ICC requirements as one of the design parameters. This layout was submitted to the Bureau of Explosives and the canister design was quickly approved as a shipping container for TMA with no modifications. GCA Corporation and Texas Alkyls, jointly, were issued ICC permit No. BA-464 for the shipment of TMA in the GCA Corporation-designed canister by either rail or motor freight. Present regulations limit the quantity of aluminum alkyls that can be shipped by Railway Express in one package at two ounces and none at all by air. The Railway Express regulation is due to be changed within the next 3 to 4 months and will allow up to 70 pounds per package to be carried in approved containers.

ASSEMBLY PROCEDURE AND SAFETY

The same precautions required for handling sodium vaporizer canisters apply to TMA canisters. One additional requirement is that personnel performing the assembly and arming operation must wear approved protective clothing designed for use by personnel handling aluminum alkyls. The clothing used by GCA Corporation personnel was purchased from Mine Safety Appliances Co. It is made of rubberized fiberglass coated with aluminum. The back of the suit, including the legs, are split. The suit is held on the wearer by sewn-in spring hoops which encircle the neck, hips, and legs. This design permits the wearer to remove the suit in a few seconds, if necessary. Figure 15 shows the protective suit. This form of protection is necessary since the reaction of aluminum alkyls with living tissue is instantaneous and extremely destructive. The ignition time when exposed to air is about 11 msec and it is very difficult to extinguish. The flame temperature is 1800°C. The use of the aluminized suit allows the wearer to have the few seconds necessary to retreat a safe distance and remove the suit before it is burned through. It cannot be too greatly emphasized that only those personnel who are thoroughly familiar with the operation should be allowed to work with the loaded canister.

The loaded canister as received at the test site does not contain the Eager-Pak pressure cylinder or the explosive valve trigger. The following assembly procedure has been developed to prepare the canister for installation on the vehicle:

IMPORTANT: Do not perform any operations on the loaded canister before becoming completely familiar with the procedures and the consequences of any errors.



Figure 15. Protective clothing.

Always wear the approved protective clothing and become adept at getting out of the clothing quickly in an emergency.

Always have an assistant (wearing protective clothing) double-checking the operation and standing by in case of emergency.

(1) Remove shipping cap including the coupling ring.

(2) Locate pressure valve, the one closest to the shell in the canister cap.

Do not touch the fill valve on the raised center portion of the cap. Opening this valve will expose TMA to the air resulting in instantaneous combustion.

(3) Check pressure valve with special wrench to make sure it is closed (clockwise); then carefully loosen knurled shipping plug on top of valve and, if there are no signs of leakage, remove knurled plug.

Do not lose the plastic washer under the plug as this is needed later.

(4) Check for bladder leakage. Using the special valve wrench, open the pressure valve slightly (counterclockwise) and be ready to react immediately should flames appear. A slight hissing sound may be heard if there is a slight differential between the pressure inside the canister and the atmospheric pressure. With the valve open three turns, wait two to three minutes to make certain everything is stable; then attach the rubber hose to the valve using the plastic washer taken from the shipping plug.

(5) Install Eager-Pak cylinder assembly and connect hose to fitting on regulator. Clamp hose to shell using clamps on hose.

(6) Install coupling ring.

(7) Install orifice to be flown.

(8) Install explosive valve trigger and tighten securely to prevent leakage after valve is fired in flight.

(9) Assemble the canister to the rest of the payload and then to the vehicle in the normal manner. The payload is now ready for arming and flight.

REMEMBER that protective clothing should still be worn during all assembly and arming operations.

CONCLUSIONS

The performance of the canister has been proven by successful completion of environmental and flight tests. The extremely dangerous properties of TMA imposed stringent design criteria on the canister development; however, these criteria make it well suited for use with other liquids including pyrophoric, corrosive, and toxic materials. The fluid capacity of the system can be altered at minimum cost by simply changing dimensions on existing manufacturing drawings.

RECOMMENDATIONS

Recommendations are made for future work to improve and further evaluate the TMA payload module.

Conduct the following:

- (1) Storage Tests: Store three canisters, filled with TMA, for a period of six months. Examine and determine whether suitable for flight.
- (2) Seals: Investigate the use of teflon seals.
- (3) Expulsion Tests: Perform additional calibrative expulsion tests under vacuum conditions to simulate flight ambient environment. Tests should be performed with the Eager-Pak pressure bottle which is used in flight.

APPENDIX A

Chemical data for TMA is given. These data were obtained from Texas Alkyls Company bulletin. Figure A-1 shows the anti-freeze property triethyl-aluminum (TEA); freezing point of the 75/25 mixture used is -40°F. The freezing point permits operational use at various launch sites.

Effects of TMA/TEA mixtures on various materials of construction are listed:

Material	Test Fluid*	Duration of Test	Comments
Silicon Rubber	A	1 day	Sample disintegrated immediately
Silicon Rubber	B	1 day	Sample disintegrated immediately
Kel-F Impregnated Glass Cloth	A	7 days	no change
Kel-F Impregnated Glass Cloth	B	7 days	no change
Polyethylene Tubing	A	7 days	slight softening
Leather Strap	B	3 to 5 days	no change

*Test Fluid "B" 75 wt. % TMA, 25 wt. % TEA
 "A" 88 wt. % TMA, 12 wt. % TEA

Chemical properties of trimethylaluminum:

Formula	$(\text{CH}_3)_3\text{Al}$
Formula weight	72.09
State and appearance at 25°C	colorless liquid
Stability:	
to air	flames instantly
to water, acids, halogens, alcohols, amines	reacts violently
Solubility	miscible with saturated hydrocarbons
Density, gm/ml at 20°C	0.752
Freezing point, °C	15

EFFECT OF TRIETHYLALUMINUM ON FREEZING POINT OF TRIMETHYLAUTUMINUM

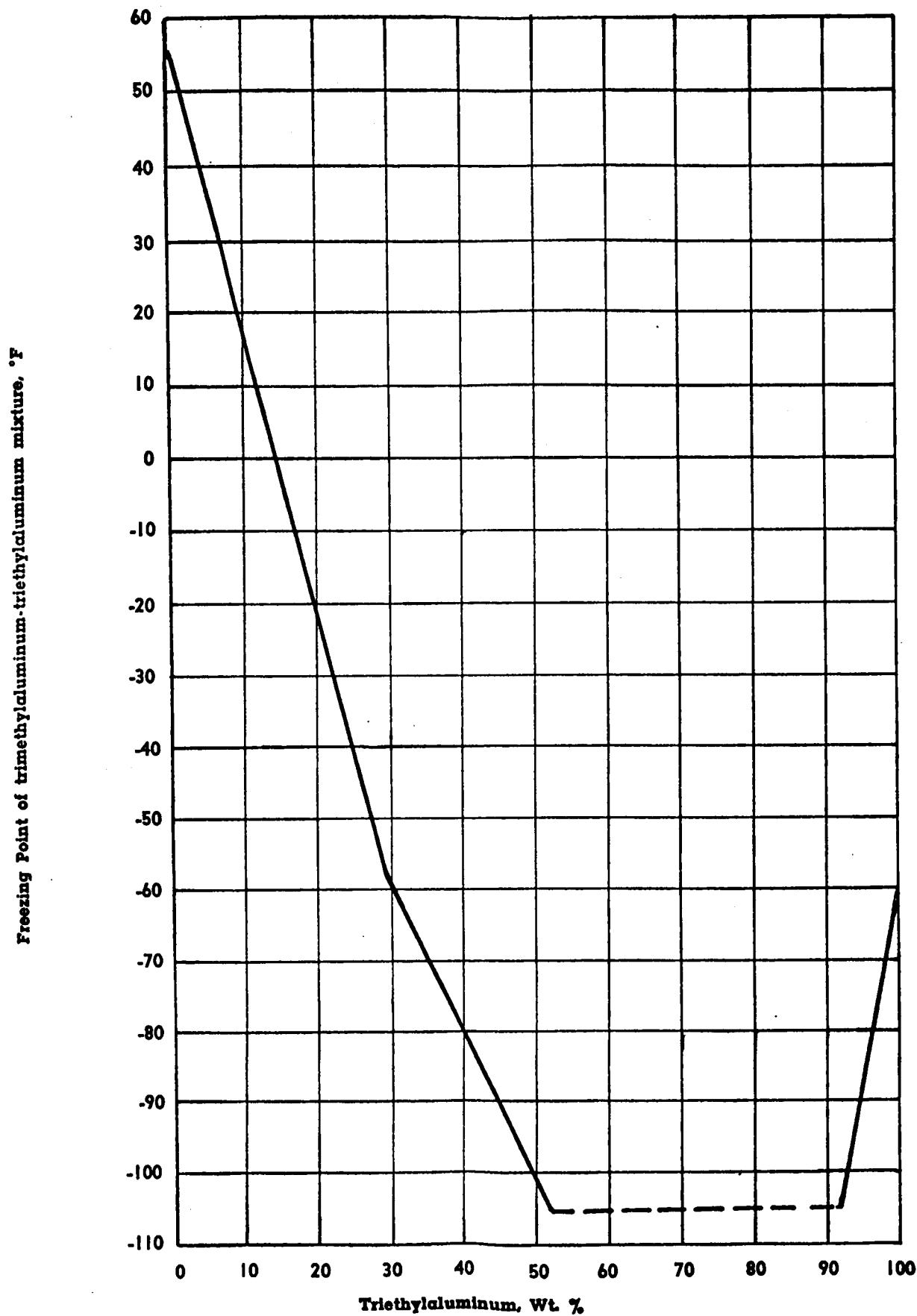


Figure A-1. Effect of TEA on freezing point of TMA.

Chemical properties of Trimethylaluminum: (continued)

Vapor pressure data:	Temp. °C	Press. mm Hg
	125-126	760
	100	332
	60	68.5
	20	8.4

Head of Combustion K cal/gal 10.5 (net)

Refractive Index n_D^{20} 1.432

Heat of formation K cal/mole 9.8

Derivation: By sodium reduction dimethylaluminum chloride

Use (Commercial): catalyst for olefin polymerization; pyrophoric fuel, manufacture of straight-chain primary alcohols and olefins.

Shipping Regulations: Flammable liquid. Red Label.*

To obtain ICC data relative to packaging, shipping, etc., write to:

Bureau of Explosives
63 Vesey Street
New York 7, New York

*Indicates "hazardous or dangerous material."

ACKNOWLEDGEMENTS

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